PROJECT NUMBER: 1754
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components
PROJECT LEADER: W. N. Einolf
PERIOD COVERED: October, 1985

MS - (D. Magin, N. Einolf)

Cigarettes made from LTF-IIA with 10% DAP did not burn. LTF-IIA filler is now being made with 5% DAP. The LTF-IIA + 10% arbutin (hydroquinone-β-D-glucopyanoside) cigarettes were smoked, the TPM extracted with CH_3CN and the extract reacted with BSTFA. Hydroquinone-TMS was the predominant smoke product, with a significant amount of the intact arbutin-TMS present (24% of the hydroquinone-TMS GC peak area).[2]

A sample of an extract from a smokeless cigarette product (FAVOR-R) was analyzed by GC/MS at the request of C. Kroustalis. Compounds identified by their mass spectrum included several hydrocarbons and related alcohols, an alkaloid with MW160, nicotine, myosmine, N-methyl-3-pyridine carboxamide, nicotine-N-oxide, nicotine-N'-oxide, nicotyrine, 4-methyl-2,6-di-tert-butyl phenol, nornicotine, cotinine, N-formyl nornicotine, and two unidentified pyridinyl ketones.[3]

Other samples analyzed by GC/MS included a mixture of four megastigmatrienone isomers (R. Southwick) two nopinone derivatives (D. Howe), a pyrazinyl propanone (D. Williams), two alkyl substituted oxoheptanals (D. Williams) and a dimethyl-α-ionone sample (F. Chappell).[3]

The transfer of the Wiley data base to the SS-200 data system is nearly complete, with about 61,000 spectra now entered.[3] Additional spectra are being entered manually by a COE student, with these spectra being added to the Flavors library.

NMR - (J. Wooten, R. Bassfield, J. Campbell)

J. Wooten made the presentation "^{13}C CP/MAS NMR Spectra of Tobacco and Tobacco Constituents" at the 5th Philip Morris Science Symposium, October 18, 1985

Structural studies of carbohydrates (with G. Chan) continue with the analysis of a dibenzoyl acetylated di-mannose compound. Proton and carbon spectra are complete. Substitution of a benzoyl group in the C-2 position produces a downfield shift in H-2, thus simplifying the overlap region (3.9-4.2 ppm) of previous compounds.[1]

NMR analysis is complete for three of the twelve nicotine analogs involved in a chemical shift study. The measurements are being made on the $N-CH_3$ and the 4α, β and 5α, β protons, and require the HOMCOR 2DJ experiment. This technique works well for the less severely overlapped resonances, but may not work adequately for other compounds.[1]

A new pulse sequence based on detecting long range proton-carbon couplings has been implemented and tested on the XL-300 NMR. This sequence may be especially useful for structural determinations of alkaloids, and is being used on model compounds.[4]

REFERENCES:

1. R. Bassfield, N.B. 7398.
2. W. N. Einolf, N.B. 8040.
3. D. F. Magin, N.B. 8222.
4. J. Wooten